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THE DETERMINATION OF THE HELIUM CONTENT OF SOME JAPANESE MINERALS.

By Jirô SASAKI.

Received November 2, 1926. Published December 28, 1926.

F. Bordas[1] has already noted the presence of helium in naegite, a radioactive mineral occurring in the Naegi district, Japan. No quantitative study on this line, however, having been made on the Japanese specimens of other minerals, so the author has undertaken the present investigation. That most minerals when simply heated evolve nearly half the amount of total helium contained, has been described by M. W. Travers,[2] and this method of extraction was adopted for the following helium determination.

The apparatus used was constructed according to the description of R. J. Strutt,[3] some parts of which were conveniently modified. A quantity of the powdered mineral was introduced in a quartzglass tube placed in an electric furnace, and heated in the exhausted state after having been washed with pure oxygen to remove all the air in the tube. The gas evolved was passed over solid potassium hydroxide, copper oxide heated to redness, and lastly over soda-lime, so as to remove most of hydrogen and carbon dioxide. The remaining gases were collected by a Toepler pump in an explosion eudiometer with platinum wires sealed in the top, and mixed with an excess of oxygen, the brush arc was then allowed to produce in the mixture by means of an induction coil to convert nitrogen into nitric oxide. The nitric acid thus formed was removed by the moistened fragments of solid potassium hydroxide floating on the surface of mercury in the eudiometer. After this operation was over, the excess of oxygen was absorbed by melted phosphorus. The gas, which was left unabsorbed and dried by phosphorus pentoxide, was introduced into a modified Geissler tube provided with electrodes of liquid alloy of potassium and sodium. Having been discharged between these electrodes, all traces of nitrogen, hydrogen, and carbon compounds were removed, and the remaining gas was finally subjected to the action of cocoanut charcoal chilled by liquid air. The gas thus having remained in apparatus was invariably pure helium which were always confirmed spectroscopically, and transferred into a narrow graduated burette where its volume was measured. The results are given in the following table.

(1) *Compt. rend.*, **146** (1908), 628.
(2) *Proc. Roy. Soc.* (*London*), A, **64** (1898), 141.
(3) *Proc. Roy. Soc.* (*London*), A, **80** (1908), 572.

Mineral	Locality	Sample taken, gr.	Helium yielded, c.c. at N.T.P.	Helium c.c. per gr. of mineral	Helium %
Fergusonite	Naegi	10.03	1.16	0.12	0.0021
Monazite	Ishikawa	17.76	2.71	0.15	0.0027
Monazite sand	Sekigammen	60.95	9.53	0.16	0.0028
Monazite sand	Shokuzan	20.21	1.91	0.095	0.0017
Beryl	Ishikawa	40.93	0.10	0.0025	0.000044
Beryl	Tanokami	20.47	No He. was detected	—	—
Beryl	Naegi	20.82	No gas was evolved	—	—

According to Prof. Y. Shibata and K. Kimura,(1) monazite of Ishikawa contains 2.91 percent of ThO_2 and 0.85 percent of UO_3, while fergusonite of Naegi 11.08 percent of ThO_2 and 0.42 percent of UO_3. Using these data, the geological ages of both minerals were calculated after the method of E. Rutherford.(2) The volumes of helium, which are to be produced per year from each gram of these two minerals are 3.4×10^{-6} c.mm. and 1.57×10^{-6} c.mm. respectively. At these rates, the time required to produce twice the quantity of helium given in the above table is 90 million years for monazite and 150 million years for fergusonite. The comparatively lower values of helium content of those minerals may thus be ascribed to their younger geological ages.

In conclusion, the author wishes to express his sincere thanks to Dr. S. Iimori for his valuable suggestions and helpful advices while performing this work ; his thanks are also due to Mr. S. Nishiyama for his assistances.

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(1) *J. Chem. Soc. Japan*, (in Japanese), **42** (1921), 1 & 957.
(2) "Radioactive Substances and Their Radiations," (1913) p. 562.

THE TRUE VISCOSITY AND THE COLLOIDAL VISCOSITY.

By **Jitsusaburo SAMESHIMA.**

Received November 12, 1926. Published December 28, 1926.

It is one of the characteristic properties of the lyophile sol that it has remarkably larger viscosity than that of the pure dispersing medium. Most of the organic "resinous" or "gelatinous" substances make viscous lyophile sols when they are brought into solutions. Cane sugar or glycerine, in the other hand, give viscous aqueous solutions though they are crystalline substances. The colloidal solutions are considered to be composed of very large "solute" particles suspended in the medium of solvent, so they may be called heterogeneous. In lyophile sols, moreover, these vulky particles have very porous structure. The viscosity of the lyophile sol is caused by the friction in moving of these vulky porous masses, or in other words friction in mass motion of solute and solvent molecules. But the viscosity of glycerine or cane sugar solution is caused by the friction between molecules of solvent or solute.

Now if we send a small particle—an ion—to travel through the liquid, then it will encounter some resistance. This resistance will be large if there are many obstacles—glycerin or sugar molecules—scattering in the way. And the resistance will be small if there are many channels of clear ways through which the particle can travel without being disturbed. The former case is assigned to the true or molecular viscosity and the latter to the colloidal viscosity. We may, therefore, clearly distinguish the two kinds of viscosities by the measurements of the electrical conductivities of the viscous solutions which contain a definite quantity of an electrolyte.

For the relation between the viscosity (η) and the conductivity, Johnstone[1] proposed the following equation,

$$\lambda = k\left(\frac{1}{\eta}\right)^m,$$

where λ is the equivalent conductivity of the solution, and k and m are constants for one kind of ion.[2] This equation may be applicable for the pure

(1) Johnston, *J. Am. Chem. Soc.*, **31** (1909), 1010.
(2) Washburn, *J. Am. Chem. Soc.*, **33** (1911), 1461 & 1686.
Sachanov, *Z. Elektrochem.*, **19** (1913), 588.

aqueous solutions of salts, but cannot be used in our experiments, viz. alcohol, glycerine or sugar solutions. In the other hand Dumanski[1] has shown that the conductivity of the solution of an inorganic salt in gelatine jelly is only slightly less than those of equally concentrated solution in pure water.

Experimental. The apparatus used is shown in accompanying figure. This apparatus has been made by inserting two platinum electrodes in an Ostwald viscosimeter. So we can measure the viscosity and the conductivity of one liquid at the same time. The concentration of the solution was chosen so as its relative viscosity was 2.5 at 25°. This can easily be attained by repeating the viscosity measurements several times for each solute. In addition to the organic matters all solutions contain 0.1 normal potassium chloride which serves as the conducting material. In each case the conductivity of the solutions of no potassium chloride was determined in order to know the correction value for the conductivity of the electrolytes which existed in the organic matter as impurities, and such a value was subtracted from the conductivity of the solution containing potassium chloride. The results are shown in the following table.

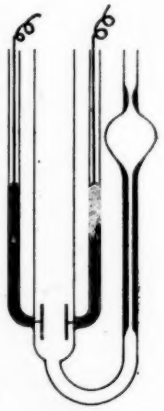

Fig. 1.

Solutes	Density d_4^{25}	Relative viscosity	Specific conductivity
KCl (0.1 N.) only	1.0019	1.005	0.01288
KCl (0.1 N.) and ethyl alcohol (ca. 50% in volume)	0.9278	2.50	0.00441
" " glycerine (ca. 35% in volume)	1.0825	2.50	0.00580
" " mannite (ca. 30% in wt., super-saturated)	1.1030	2.50	0.00621
" " cane sugar (ca. 30% in weight)	1.1160	2.50	0.00654
" " gum arabic (ca. 5.5% in weight)	1.0208	2.50	0.01160
" " soluble starch (ca. 1% in weight)	1.0052	2.43	0.01235
" " gelatine (ca. 3.4% in weight)	1.0113	2.47	0.01286

From the above table we see, in general, that the heavy solute molecules give less effect on conductivity than the light molecules, provided the viscosity being the same. Of course there is a great effect of solvent on dissociation degree of electrolyte, so the conductivity is influenced not only by the viscosity but also by the dissociation degree. Among the colloidal solutions gum arabic give stronger effect than gelatine, consequently the former may have lighter " molecular weight " than the latter,

(1) Dumanski, *Z. physik. Chem.*, **60** (1907), 553.

Summary.

There are two kinds of viscosities, viz. the true viscosity and the colloidal viscosity. They can be distinguished by the conductivity measurements. The true viscosity give strong effect on the conductivity but the colloidal viscosity give slight effect on it.

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ON THE DERIVATION OF ADSORPTION ISOTHERMS. II.

By **Bun-ichi TAMAMUSHI.**

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In the previous work[1] the author has remarked that an exponential adsorption isotherm $C=ku^i$ could be derived from the equation of Gibbs, if we assume at the same time, the following relation,

$$(\gamma_0-\gamma)\frac{1}{u}=iRT \quad \cdots\cdots(1)$$

in which $(\gamma_0-\gamma)$ is the difference between the surface tension of solvent and that of solution, u the excess of solute per unit area in the surface, R the gas constant, T the absolute temperature and i a certain constant, the physical meaning of which remains to be explained.

Now the diminution in the surface tension by the adsorption film can be put equal to the pressing force P_s, across unit length of the boundary, and u the reciprocal of the adsorption surface A where the adsorption of one mol of solute takes place. So that equation (1) will be written down as follows, the correction i being dropped,

$$P_sA=RT \quad \cdots\cdots(2)$$

This is known as the equation for the so-called "gaseous film," and it holds when the solution is sufficiently dilute and the average kinetic energy in each degree of freedom in the surface, $\frac{1}{2}RT$ for each molecule, acts wholly to produce the pressing force on the boundary. When the solution becomes more concentrated, due corrections are necessary. In this respect, the intermolecular forces are to be taken into account in the first place, so that we have the following relation instead of equation (2):

$$\{P_s-f(E)\}A=RT \quad \cdots\cdots(3)$$

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(1) Tamamushi, this journal **1** (1926), 185.

where $f(E)$ is a certain function which represents cohesion or repulsion between molecules. Certainly it is difficult to give this function a complete elucidation. At any rate, we can probable consider that molecules are electric dipoles and that there takes part potential energy of electric nature in the surface besides the thermal kinetic energy. Here let us assume that the polarization of dipole molecules in the surface is proportional to $\sqrt{A}$ i.e. to the mean distance between the molecules. Now, one end of the dipole molecules being attracted by the main body of the solution, the charge of the same sign will be arranged in the surface, the effect of which weakens the surface attraction. Let us further assume that the probability of a molecule in taking proper orientations fully to display this effect of polarization is proportional to $\sqrt{T}$, in other words, to the mean velocity of the molecule. The repulsive effect of a molecule will then be proportional to $\sqrt{A}\cdot\sqrt{T}$, and consequently the repulsive force between adjacent molecules can be represented by $q\frac{(\sqrt{A}\cdot\sqrt{T})^2}{A^2}$ or by $q\frac{T}{A}$, where q is a proportional constant.

If this expression is put into equation (3) in place of $f(E)$, the following relation will be obtained,

$$\left(P_s-\frac{qT}{A}\right)\cdot A=RT$$

or

$$P_s\cdot A=RT+qT$$

And if we put here

$$1+\frac{q}{R}=i \qquad \cdots\cdots(4)$$

we have

$$P_s\cdot A=iRT.$$

This is no other than equation (1).

Taking now Volmer's surface correction β into account and using the notations already mentioned, we obtain.

$$(\gamma_0-\gamma)\left(\frac{1}{u}-\beta\right)=iRT \qquad \cdots\cdots(5)$$

as a general form of the characteristic equation of the surface.

Eliminating γ from this equation and the equation of Gibbs introduced with the surface correction β i.e.

$$\frac{u}{1-u\beta}=-\frac{C}{RT}\frac{d\gamma}{dC}$$

we obtain a new adsorption isotherm as follows:

$$\frac{u}{1-u\beta}=KC^{\frac{1}{i}} \qquad \cdots\cdots(6)$$

in which K and i are constants.

This formula is identical with that of Freundlich when u is small and it also expresses the state of saturation.

An example of the application of this isotherm for experimental data is given in the following. The data here referred are taken from one of the series of experiments made by Schmidt[1] on the adsorption of acetic acid solution by animal charcoal. For the convenience sake of calculation, the formula (6) is transformed into

$$\log\left(\frac{1}{u}-\frac{1}{S}\right)=\frac{1}{i}\log\left(\frac{1}{C}\right)-\log K \cdots\cdots\cdots\cdots (7)$$

in which $\frac{1}{S}=\beta$, S representing the amount of solute in saturation. And u is assumed to be proportional to the adsorption per unit mass of solvent.

Fig. 1.

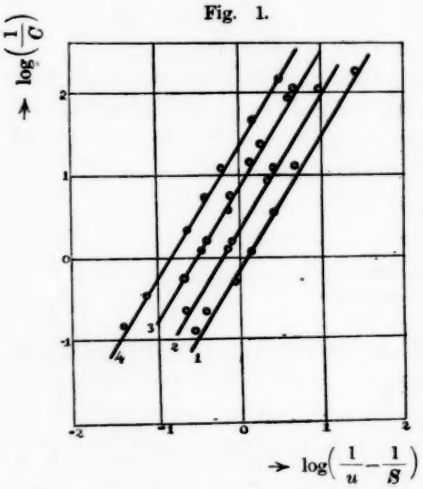

TABLE 1. 5 gr. Animal Charcoal.

$\log\frac{1}{C}$	$\log\left(\frac{1}{u}-\frac{1}{S}\right)$	$C_{obs.}$	$C_{calc.}$
$\bar{1}.12188$	$\bar{1}.54637$	7.553	11.07
$\bar{1}.37469$	$\bar{1}.57519$	4.220	6.898
$\bar{1}.69272$	$\bar{1}.95665$	2.029	1.510
0.07058	0.14259	0.850	0.720
0.53611	0.41380	0.291	0.244
1.12148	0.69346	0.0756	0.0751
2.20761	2.42586	0.0062	0.0043
$S=0.523$	$i=1.73$	$K=0.86$	

TABLE 2. 10 gr. Animal Charcoal.

$\log\frac{1}{C}$	$\log\left(\frac{1}{u}-\frac{1}{S}\right)$	$C_{obs.}$	$C_{calc.}$
$\bar{1}.34008$	$\bar{1}.39445$	4.570	4.650
0.10237	$\bar{1}.90795$	0.790	0.774
0.17393	$\bar{1}.92788$	0.670	0.555
0.95078	0.34439	0.112	0.106
1.11238	0.41731	0.0772	0.0789
2.03152	1.00689	0.0093	0.0076
$S=1.058$	$i=1.73$	$K=1.7$	

TABLE 3. 20 gr. Animal Charcoal.

$\log\frac{1}{C}$	$\log\left(\frac{1}{u}-\frac{1}{S}\right)$	$C_{obs.}$	$C_{calc.}$
$\bar{1}.76700$	$\bar{1}.38453$	1.710	1.466
0.11930	$\bar{1}.57634$	0.759	0.682
0.29826	$\bar{1}.64972$	0.5032	0.5087
0.56019	$\bar{1}.81558$	0.2753	0.2627
0.73731	$\bar{1}.90956$	0.1831	0.1807
1.18442	0.10789	0.0854	0.0819
1.40561	0.28670	0.0393	0.0435
1.97551	0.59051	0.01058	0.01198
2.04096	0.65591	0.00910	0.00923
$S=2.119$	$i=1.73$	$K=3.4$	

(1) Schmidt, *Z. physik. Chem.* **77** (1911), 645.

TABLE 4. 40 gr. Animal Charcoal.

$\log\frac{1}{C}$	$\log\left(\frac{1}{u}-\frac{1}{S}\right)$	$C_{obs.}$	$C_{calc.}$
$\bar{1}.21746$	$\bar{2}.61278$	6.06	8.50
$\bar{1}.56177$	$\bar{2}.89873$	2.74	2.72
$\bar{1}.69486$	$\bar{1}.02160$	2.02	1.67
0.36005	$\bar{1}.39410$	0.437	0.378
0.76447	$\bar{1}.62024$	0.172	0.158
1.12726	$\bar{1}.79078$	0.0746	0.0778
1.69680	0.17638	0.0201	0.0225
2.18046	0.51068	0.0066	0.0104
$S=4.434$	$i=1.73$	$K=7.1$	

Fig. 1. shows that the linear relation between $\log\left(\frac{1}{u}-\frac{1}{S}\right)$ and $\log\left(\frac{1}{C}\right)$ is satisfactory. i Is found to be fairly constant for varying amounts of adsorvent and approximately equal to 1.73. The value of K found is almost proportional to the amount of charcoal taken. In the last two columns of each table the values of concentration C (gr. in 100 c.c.) are compared with that which are calculated by relation (7).

The present note, although still incomplete, is published, the author being obliged to put off further investigations for few months. In conclusion, the author wishes to express his sincere thanks to Prof. M. Katayama for his kind guidance.

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SPECTROCHEMICAL STUDIES OF HYDROXYAZO-COMPOUNDS.[1] PART I.

By **Taku UEMURA, Naomichi YOKOJIMA,** and **Chinyü TAN.**

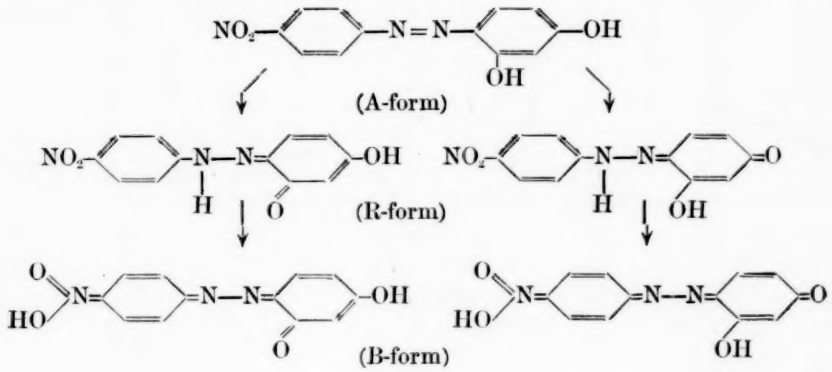
Received November 3, 1926. Published December 28, 1926.

Introduction. The authors have studied on the relation between the colour of hydroxyazo-compounds and their chemical constitution by the spectrochemical method.

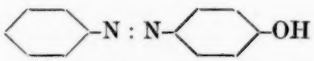
Hewitt,[2] Baly,[3] and Sircar[4] already chemically investigated some hydroxyazo-compounds with regard to their chemical constitution, and

(1) Read before the Chemical Society of Japan, October 9, 1926.
(2) Hewitt and Mitchell, *J. Chem. Soc.*, 91 (1907), 1251.
Hewitt and Fox, *J. Chem. Soc.*, 93 (1908), 333.
Hewitt and Thomas, *J. Chem. Soc.*, 95 (1909), 1292.
Hewitt and Thole, *J. Chem. Soc.*, 95 (1909), 1393; 97 (1910), 511.
(3) Baly, Tuck, and Marsden, *J. Chem. Soc.*, 97 (1910), 1497.
(4) Sircar, *J. Chem. Soc.*, 109 (1916), 757.

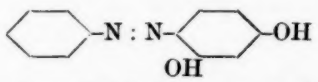
Tuck[1] did it spectrochemically. They all recognised the tautomeric changes in these compounds. K. Suitsu[2] recently observed three different colours of para-red (p-nitrobenzeneazo-β-naphthol) by changing conditions, and he respectively assigned the azo-, monoquinonoid- and diquinonoid-form to the three tautomers. We also have been able to observe the remarkable analogy in many simpler hydroxyazo-compounds and have then given the following forms respectively:



Experimental.

1. p-Hydroxyazobenzene,[3] 

A dilute neutral alcoholic solution of the substance is yellow, and changes into a deeper shade by adding a KOH-solution. As the absorption curve of its N/10000 solution shows, the bathochromic and hyperchromic influences are easily observed by the addition of alkali (Fig. 1). We give A-form to the neutral solution and R-form to the alkaline.

2. Benzeneazoresorcinol,[4] 

Bathochromic and hyperchromic effects due to alkali are also seen in Fig. 2, and the curve analogous to that of No. 1 compound (Fig. 1) was obtained. A- and R-forms can then be respectively assigned to neutral and alkaline solutions.

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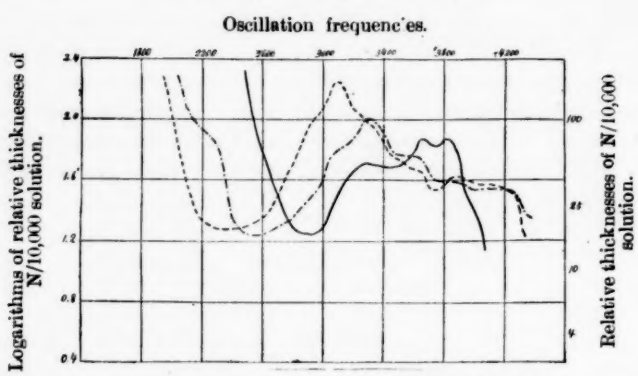
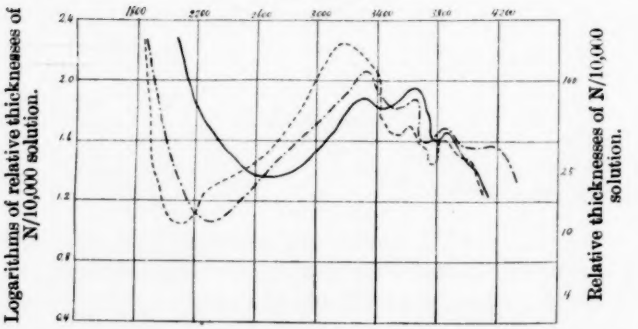
(1) Tuck, *J. Chem. Soc.*, 91 (1907), 449; 95 (1909), 1809.
(2) Suitsu and Okuma. *J. Soc. Chem. Ind. Japan*, 29 (1926), 132.
(3) Beilstein, "Handbuch der Organischen Chemie," IV (1899), 1407.
(4) Ibid., IV (1899), 1441.


Oscillation frequenc'es.
Logarithms of relative thicknesses of N/10,000 solution.
Relative thicknesses of N/10,000 solution.

1. p-Hydroxyazobenzene. { —— Neutral Solution. ----- 0.9 N. Alkaline Solution.
4. p-Nitrobenzeneazophenol. -·-·- Neutral Solution.

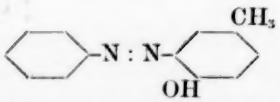
Fig. 1.


Oscillation frequencies.
Logarithms of relative thicknesses of N/10,000 solution.
Relative thicknesses of N/10,000 solution.

2. Benzeneazoresorcinol. { —— Neutral Solution. ----- 0.9 N. Alkaline Solution.
5. p-Nitrobenzeneazoresorcinol. -·-·- Neutral Solution.

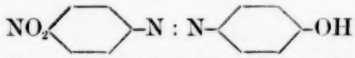
Fig. 2.

3. Benzeneazo-p-cresol,[1]

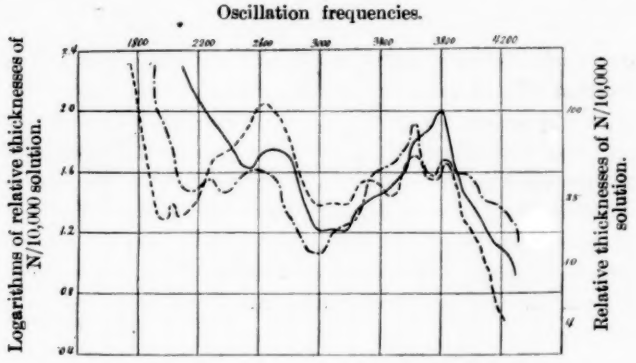


By adding KOH, the yellow neutral solution of this compound changes into red, but not blue. Two absorption bands (Fig. 3) can be observed in its neutral solution and they become clearer when we add KOH to it. We can give A-form to the yellow solution and R-form to the red one.

(1) Beilstein, "Handbuch der Organischen Chemie," IV (1899), 1420.

4. p-Nitrobenzeneazophenol,[1] 

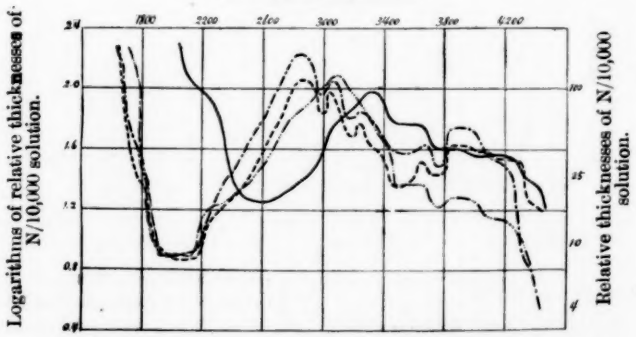
This substance undergoes quite the same change in colour as the above-mentioned compounds and like them cannot obtain blue tautomer. Hewitt[2] once gave B-form to the red solution of this compound, but according to our examination we can only give R-form to this red solution, for we can ascertain that the curve of this alkaline solution coincides to that of the red one of m-nitrobenzeneazoresorcinol (No. 6 compound) which cannot take B-form (Fig. 4).


Oscillation frequencies.
Logarithms of relative thicknesses of N/10,000 solution.
Relative thicknesses of N/10,000 solution.

3. Benzeneazo-p-cresol.
7. p-Nitrobenzeneazo-p-cresol.

Neutral Solution.
0.9 N. Alkaline Solution.
Neutral Solution.

Fig. 3.

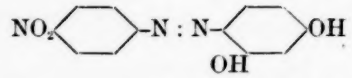

Oscillation frequencies.
Logarithms of relative thicknesses of N/10,000 solution.
Relative thicknesses of N/10,000 solution.

4. p-Nitrobenzeneazophenol.
6. m-Nitrobenzeneazoresorcinol.

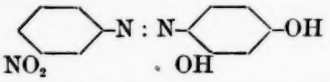
Neutral Solution.
N/500 Alkaline Solution.
1.44 N. Alkaline Solution.
2 N. Alkaline Solution.

Fig. 4.

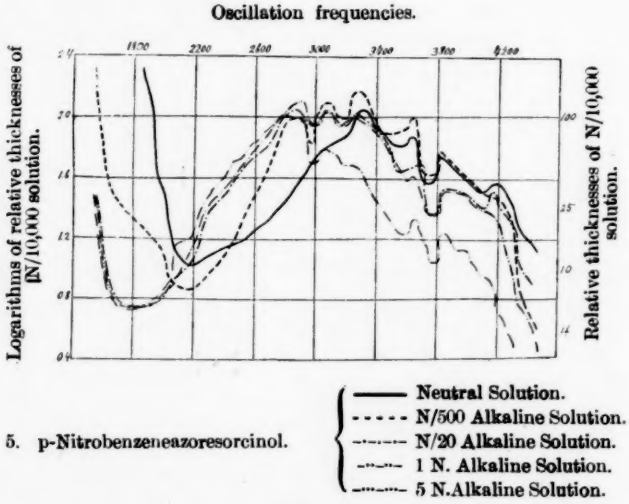
(1) Ibid., IV (1899), 1410.
(2) Hewitt and Mitchell, *J. Chem. Soc.*, 89 (1906), 17.

5. p-Nitrobenzeneazoresorcinol,[1] 

The reddish yellow neutral solution becomes red when it is N/500 alkaline solution, and bluish violet by adding a stronger alkali. Then we may respectively assign A-, R- and B-forms to these three tautomers (Fig. 5).

6. m-Nitrobenzeneazoresorcinol,[2] 

The curve of N/500 alkaline solution of No. 5 compound and that of the alkaline solution of this substance nearly coincide and, therefore, this substance has A- and R-forms (Fig. 6). This compound has no B-form, for the nitro-group, in this substance takes the meta-position with respect to the azo-group, and cannot, of course, be of the isonitroform. As the three compounds (No. 2, No. 5 and No. 6), already described, have two hydroxyl-groups (ortho and para with respect to the azo-group), we may suppose two cases of tautomeric changes, but cannot yet definitely say whether it is the hydrogen atom in para hydroxyl that moves or the one in the ortho hydroxyl.



5. p-Nitrobenzeneazoresorcinol.

Fig. 5.

(1) Beilstein, "Handbuch der Organischen Chemie," IV (1899), 1442.
(2) Obtained from m-nitraniline and resorcinol.

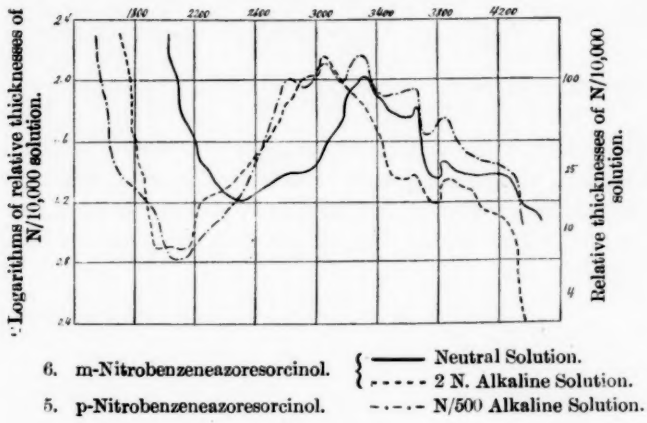
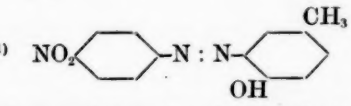


Fig. 6.

7. p-Nitrobenzeneazo-p-cresol,[1] 

The yellow neutral solution changes into red and instantly into violet by adding KOH. N/1000 alkalien solution shows already a bluish violet shade. The curve of this compound (Fig. 7) has two absorption bands like that of

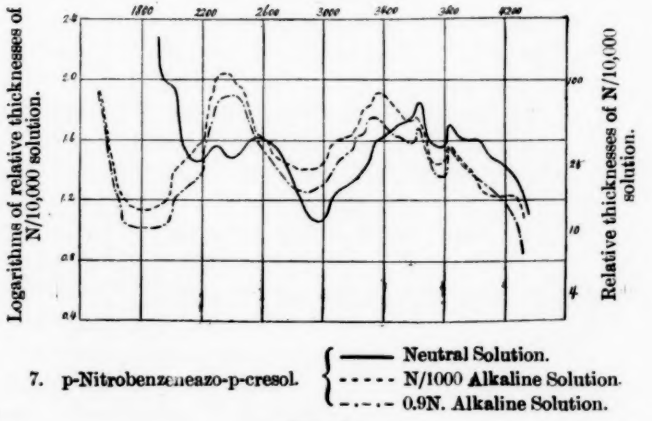
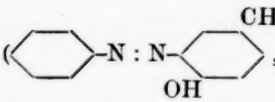
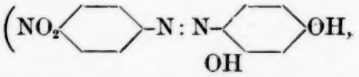


Fig. 7.

(1) Beilstein, "Handbuch der Organischen Chemie," IV (1899), 1041.

No. 3 compound ( , in Fig. 3). The absorption curve of 1 N. alkaline solution of No. 5 compound ( , in Fig. 5) to which was assigned B-form, nearly coincides to that of 0.9 N. alkaline solution of this compound, which is only more hypochromic than the former, and we have given A- and B-forms to this compound according to the condition. Of course, we may recognise the existence of R-form which is very unstable.

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